Third Quarterly Report

on

STUDY OF USE OF AUXILIARY ELECTRODES IN SILVER CELLS

1 August, 1964 - 1 November, 1964

Contract No. NAS 5-3669

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Prepared by

General Electric Company
Advanced Technology Laboratories
Schenectady, New York

for

Goddard Space Flight Center
Greenbelt, Maryland

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SUMMARY

The primary objective of this program is to investigate the use of the auxiliary electrodes in primary and secondary silver-zinc cells and to modify commercially available silver-zinc cells by addition of auxiliary electrodes for testing by NASA-Goddard. Additional work areas include the continuation and completion of testing of auxiliary electrode silver-cadmium cells remaining from Contract NAS 5-2817 and the investigation of electrochemical techniques for recombination of hydrogen evolved from primary silver-zinc cells used in satellite power supply systems. These tasks are itemized on Table I.

Project work for this quarter included the continued testing of several types of auxiliary electrodes for oxygen recombination; testing of auxiliary electrode containing platinum metal catalysts for hydrogen recombination; testing the auxiliary electrodes for their discharge characteristics against the cell electrodes; construction of a number of silver-cadmium cells with conventional auxiliary electrodes; testing of silver-cadmium cells containing auxiliary electrodes on continuous discharge; placing six of these before mentioned cells on a cycle test; and constructing several hydrogen combination cells for use in removing hydrogen from enclosed spaces.

The silver-cadmium cells on continuous overcharge showed a small initial rtse in pressure which has been followed by a decrease in pressure to an equilibrium value. These cells are on continuous charge and will be checked for charge content prior to termination of the contract. Since the auxiliary electrode is not connected, it seems that the catalytic effect of the electrode is enough to prevent pressure rise.

A new group of silver-cadmium cells containing auxiliary electrodes were placed on cycling tests. At the beginning of this test, the cycling equipment failed and the cells were discharged without charging, and gassing occurred. The equipment has been repaired and after the cycling for a month, the capacity of these cells will be determined. Following this, the auxiliary electrodes will be connected and the charging continued until the end of the contract. At that time the capacity of the cells will again be determined.

The test results on the use of water-proofed, silver-powder, auxiliary electrodes for oxygen recombination show that these electrodes are suitable for use in silver-zinc cells. However, these electrodes have to be used at a lower current density than the conventional silver electrodes containing platinum metal catalysts. An effective auxiliary electrode was made from silver powder combined with a spinel structure cobalt aluminate.

Studies showed that the silver spinel auxiliary electrode would self-discharge the zinc electrode directly to it. However, if the electrode is connected through a diode, a method patented by Ruetschi, these electrodes will pass current only in the presence of oxygen.

Test runs on auxiliary electrodes for hydrogen recombination were inconclusive because of difficulties in obtaining electrodes with suitable catalytic activity.

Seven hydrogen combination cells of two designs were constructed and tested. The cathode material was cupric oxide in both designs. The anode was either a conventional porous gas diffusion type electrode containing platinum or a solid palladium-silver alloy foil. For the present application the porous gas diffusion electrode is preferred because of its higher current capability.

TABLE I

Program Tasks

Task I - Silver-Cadmium Cells

- A. The 5.0 amp-hr test cells from the previous contract (or new ones) with auxiliary electrodes will continue to be cycled at room temperature with periodic monitoring of cell capacity and continuous monitoring of cell pressure.
- B. Five 5.0 amp-hr test cells from the previous contract with auxiliary electrodes will be continuously charged at an average potential of 1.52 volts/cell, the cell pressure and voltage being monitored.

Task II - Silver-Zinc Cells

Perform studies on the use of auxiliary electrodes for recombination of hydrogen and oxygen as a function of operating parameters to obtain design data to modify commercial silver-zinc cells of 3.0 amp-hr capacity.

- A. Study the effect of auxiliary electrode materials on the self-discharge of the zinc electrode in both the electrically coupled and uncoupled condition.
- B. Study recombination of hydrogen during charge, stand, and discharge by an auxiliary electrode connected to the positive cell electrode.
- C. Study the recombination of oxygen during charge and stand on an auxiliary electrode when connected to the negative cell electrode.
- D. Study the effect of silver and zincate ions on the hydrogen and oxygen recombination by auxiliary electrodes.

Task III - Modification of Silver-Zinc Cells

Incorporate auxiliary electrodes into fifty 3.0 amp-hr commercially available silver-zinc cells.

Task IV - Hydrogen Combination Cell

Investigate the feasibility of electrochemical recombination of hydrogen evolved from silver-zinc satellite power supply systems. Cells employing AgO and CuO will be investigated initially.

1.0 INTRODUCTION

This report covers the work done during the third quarter of a twelve-month program to investigate the use of auxiliary electrodes in silver-zinc cells. Two additional work areas are the continuation and completion of cyclic test of silver-cadmium cells with auxiliary electrodes from a previous contract (NAS 5-2817) and the investigation of the feasibility of combining hydrogen evolved from a silver-zinc satellite power supply system by means of an electrochemical cell.

Of primary concern, in the application of auxiliary electrodes to silver-zinc cells, is poisoning the zinc electrode with platinum from the auxiliary electrode which catalyzes the selfdischarge reaction of the zinc electrode:

$$Zn + 2 KOH \longrightarrow K_2 ZnO_2 + H_2$$

It is believed that the danger of platinum migration to the zinc electrode is small since, 1) the binder of the electrode has been very effective in holding the catalyst in place and has shown no deterioration in electrodes used for 10-14 months; 2) the platinum is in a highly insoluble form and has shown no tendency to go into solution when the electrode is used at current densities below 50 ma/cm²; 3) the barriers used in silver cells for blocking silver migration are equally effective for platinum, since platinum compounds are more easily reduced or absorbed than silver.

Although the auxiliary electrode has been used primarily as an oxygen electrode in silver-cadmium applications, there is no basic reason for an auxiliary electrode being operated only as an oxygen electrode. It can also be operated as a hydrogen electrode. For use as a hydrogen electrode, the auxiliary electrode would be connected to the positive (silver oxide) electrode in use, the recombination reactions being:

$$H_2 + 2OH^- \longrightarrow 2 H_2O + 2 e$$
 $Ag_2O + H_2O + 2 e \longrightarrow 2 Ag + 2 OH^-$

With this type of auxiliary electrode operation, the cell should be designed to be negative plate limiting, so that hydrogen is preferentially evolved on charging. The use of a hydrogen recombination system is desirable for silver-zinc cells, since there is an evolution of hydrogen during the cell stand and discharge that leads to pressure build-up in many applications.

The use of two auxiliary electrodes -- one for oxygen recombination which is attached to the negative electrode, and one for the recombination of hydrogen which is attached to the positive electrode -- is possible. The principal advantage of this dual system is that gassing of either hydrogen or oxygen would be taken care of. Also, the use of a platinum containing catalyst for the oxygen electrode is not needed thus removing the possibility of zinc electrode degradation by coupling through the external circuit. This non-platinum type of electrode was originally proposed for use in silver-cadmium cells, but was abandoned in favor of the currently used one which can recombine small amounts of hydrogen directly, since this property was useful and presented no problems in use in silver-cadmium cells.

Another potential problem area is the possible poisoning of the auxiliary electrodes by interactions with both silver and zincate ions present in the electrolyte phase of these cells. At present, there is no evidence that either will degrade the electrode for oxygen or hydrogen recombination. This aspect, however is being investigated.

An outline of the program work content by tasks is given in Table I.

2.0 WORK ACCOMPLISHED

The work during the third quarter was largely concerned with carrying out the initial parts of the work program established. The accomplishments of the quarter are discussed by tasks in the following sections.

2.1 Silver-Cadmium Cells

The two types of tests were performed with silver-cadmium cells. Cells were run at continuous overcharge to determine that if hydrogen is generated in this mode of operation, it will catalytically recombine with oxygen. Other silver-cadmium cells were continuously cycled to determine the influence of this type of operation on performance.

2.1.1 Continuous Charge

The purpose of this test is to determine the behavior of the cells on continuous overcharge such as would be experienced on satellites in a polar orbit. For the initial part of the experiment, the auxiliary electrode is not connected with either electrode in the cell; this is to test the hypothesis that only a gas recombination catalyst is needed for cell operation under constant voltage charging to prevent excessive buildup of gas pressure or loss of cell balance.

Table II shows the results of the overcharging tests (including the first two weeks that were reported in the second quarterly report). The voltage of the auxiliary electrode is also monitored daily, since the sign and the magnitude of the potential is an indication whether excess oxygen or hydrogen is present. As of November 1, only oxygen was indicated in the test cells. The continuous charging tests will be interrupted in January to allow checking of the capacity of the test cells.

In the first month, the pressure in all the cells originally increased to a maximum up to 26 psi in one of the cells. After attaining a maximum pressure, the cell pressures decreased slowly with a minimum, all the cells currently operating at a pressure less than atmospheric.

2.1.2 Cycling Tests

New test cells were fabricated from Yardney YS-5 electrode packs consisting of four silver and five cadmium electrodes and the original separators. The auxiliary electrode was of the

TABLE IIa
Continuous Charge (1) Cell Data

Cell #	061501	061502	061503	061504	061505	061506
Day			Pressure (2))		
7/20 7/21 7/22 7/23 7/24	30 in. 30 in. 25 in. 5 in. 3 psi	30 in. 30 in. 28 in. 26 in. 25 in.	25 in. 15 in. 5 psi 12 psi 9 psi	18 in. 5 in. 5 in. 5 in. 5 in.	4 psi 10 psi 20 psi 23 psi 23 psi	30 in. 23 in. 20 in. 15 in. 12 in.
7/27 7/28 7/29 7/30 7/31	8 psi 9 psi 11 psi 12 psi 13 psi	18 in. 12 in. 3 in. 2 psi 3 psi	6 psi 8 psi 5 psi 4 psi 2 psi	l psi 2 psi 0 psi 2 psi 1 psi	17 psi 15 psi 14 psi 14 psi 12 psi	7 in. 5 in. 5 in. 4 in. 0 in.
8/3	14 psi	4 psi	3 psi	0 psi	8 psi	4 psi
8/7	17 psi	8 psi	3 psi	l psi	7 psi	6 psi
8/13	18 psi	9 psi	2 psi	l psi	5 psi	5 psi
8/19	15 psi	8 psi	2 psi	l psi	2 psi	3 psi
8/25	10 psi	4 psi	0 psi	0 psi	0 psi	2 psi
9/3	l psi	5 in.	6 in.	4 in.	8 in.	4 in.
9/10	7 in.	ll in.	12 in.	4 in.	9 in.	5 in.
9/16	16 in.	17 in.	13 in.	4 in.	9 in.	4 in.
9/22	21 in.	20 in.	12 in.	3 in.	7 in.	0 in.
9/28	22 in.	21 in.	12 in.	3 in.	5 in.	0 in.
10/2	22 in.	21 in.	8 in.	2 in.	3 in.	0 in.
10/8	20 in.	22 in.	8 in.	2 in.	l in.	0 in.
10/14	19 in.	20 in.	5 in.	l in.	0 in.	0 in.
10/20	18 in.	20 in.	5 in.	l in.	0 in.	0 in.
10/26	16 in.	20 in.	5 in.	0 in.	0 in.	0 in.
10/30	16 in.	20 in.	5 in.	0 in.	0 in.	0 in.

⁽¹⁾ Constant voltage, 9.12 + 0.06 volts on battery (1.52 \pm 0.01 volt/cell avg). Started 8 a.m. 7/20/64, readings taken at 4 p.m. each day.

⁽²⁾ Pressure in inches of vacuum or guage PSI.

TABLE IIb

Continuous Charge Cell Data

Voltage (3)

	Cell #	061501	061502	061503	061504	061505	061506	Charge (4)
	<u>Day</u>							
	7/20 7/21 7/22	1.48 1.51 1.54	1.50 1.53 1.52	1.50 1.54 1.56	1.51 1.51 1.50	1.72 1.60 1.58	1.50 1.49 1.48	60 30 16
	7/23 7/24	1.56 1.56	1.52 1.52	1.56 1.56	1.50 1.53	1.56 1.56	1.48 1.48	6 4
	7/27 7/28 7/29 7/30 7/31	1.54 1.54 1.53 1.53 1.54	1.54 1.53 1.53 1.53 1.53	1.54 1.54 1.54 1.53 1.54	1.54 1.53 1.53 1.53	1.55 1.54 1.54 1.54 1.53	1.49 1.48 1.51 1.53 1.54	3 3 3 3 3
	8/3	1.54	1.53	1.54	1.53	1.54	1.53	3
	8/7	1.54	1.53	1.54	1.54	1.54	1.54	3
	8/13	1.53	1.53	1.54	1.54	1.54	1.54	3
•	8/19	1.54	1.53	1.54	1.55	1.55	1.55	3
	8/25	1.53	1.53	1.53	1.53	1.54	1.56	3
()	9/3	1.51	1.53	1.52	1.53	1.54	1.65	2
	9/10	1.51	1.51	1.51	1.51	1.54	1.68	2
	9/16	1.47	1.49	1.49	1.50	1.54	1.69	1.5
	9/22	1.46	1.48	1.46	1.47	1.63	1.65	1
	9/28	1.46	1.48	1.46	1.47	1.64	1.65	1
	10/2	1.46	1.46	1.45	1.46	1.65	1.64	1
	10/8	1.47	1.48	1.46	1.47	1.66	1.66	ı
	10/14	1.46	1.47	1.46	1.47	1.65	1.63	1
	10/20	1.46	1.46	1.45	1.46	1.64	1.63	1
	10/26	1.47	1.47	1.46	1.47	1.66	1.65	1
	10/30	1.45	1.46	1.45	1.46	1.64	1.63	1

⁽³⁾ Volts/cell using 1000 ohms/volt meter.

⁽⁴⁾ Charge current, milliamperes.

same material used previously, i.e. a nickel wire mesh coated with a thin layer of electrode catalyst containing platinum metals, plus a nickel powder electron collector and Teflon binder. dimensions of the auxiliary electrode are the same as the dimensions of the YS-5 electrodes. The auxiliary electrode is placed against one of the two end cadmium electrodes with a Pellon 2505W Nylon separator between the two. The gas side of the auxiliary electrode is separated about 3/16 inch from the cell case by a piece of polyethylene grid (Vexar Plastic Netting, Vexar Division, E. I. du Pont de Nemours, Buffalo, N. Y. diamond pattern 4/4-30-90-PE-3). An additional 1/4 inch length of cell case has been provided to serve as electrolyte storage during cycling operation; wicks of Nylon separator were placed to extend from the bottom of the original cell separators to the bottom of the reservoir. The test cells are potted in 1 inch of epoxy and are fitted with a pressure connection to a pressure transducer. A toggle valve is provided to permit connection of the gas system to external gas pumps or analysis equipment as desired.

The test cells were formed by completely filling the cell with 30% potassium hydroxide and allowing a 24 to 48 hour soak period to equilibrate the separators, then charging for 20 hours at 300 ma. constant current. The electrolyte level was then adjusted so that the bottom of the electrode pack was immersed in 1/8 inch of electrolyte. The cell was discharged at 2.5 amperes to a 1.0 volt cut off. The electrolyte level at the end of discharge was about 1/8 inch deep in the reservoir; this assures that the electrode pack separators are always wetted.

These cells were cycled at the following rate:

Discharge - 1/2 hour at 2.5 amperes Charge - 11-1/2 hours at 0.12 amperes

During this cycling, cell potentials, auxiliary electrode versus cadmium voltage and the pressure, and auxiliary electrode current were also recorded. During the preliminary cycling, the cycling equipment failed and the cells were discharged several times without charging. This produced gassing within the cells. The equipment was repaired and the cells have been cycled the last month. The capacity of these cells will now be determined and further cycling will be continued with the third electrode properly hooked up in the cell.

2.2 Silver-Zinc Cells

This task is concerned with obtaining design data on the suitability of various auxiliary electrode compositions for the hydrogen and oxygen recombination. Factors which will be evaluated are: recombination of oxygen and hydrogen at various levels of gas pressure, effect of auxiliary electrode material on self-discharge on zinc electrodes, and the effect of silver and zinc ions on the rate of recombination.

Several types of auxiliary electrodes were used in these experiments. For oxygen recombination, the following types were used:

<u>Silver mesh</u> - Expanded silver metal mesh (AG-079-2) manufactured by Designers Metal Corporation.

<u>Silver powder</u> - Handy and Harmon Silflake 131 silver powder pressed on a silver screen into sheets with Teflon as a waterproofing and binding agent.

<u>Silver spinel</u> - Handy and Harmon Silflake 131 silver powder and a spinel structure cobalt aluminate pressed on a silver screen into sheets with Teflon as a waterproofing and binding agent.

For hydrogen recombination, the following types were used:

<u>Conventional</u> - A mixture of nickel, platinum and palladium pressed into sheets on a nickel mesh with Teflon as a water-proofing and binding agent.

<u>Silver-Modified Conventional</u> - A mixture of silver-platinum and palladium pressed into sheets on a silver mesh with Teflon as a waterproofing and binding agent. This electrode is similar to the one described above except that silver is used in place of nickel.

2.2.1. Self-discharge of Zinc Electrodes

The composition of conventional auxiliary electrodes for oxygen recombination was found to be unsatisfactory for this function in silver-zinc cells. The platinum contained in these electrodes promotes a rapid discharge with the zinc electrode electrically connected through a load resistor.

The silver mesh, silver powder, and silver spinel electrodes (electrode are 14 cm²) were connected to a zinc electrode from a three ampere hour Yardney silver zinc cell (Model PM-3) and the current through a l ohm load measured. The gas formed was collected at the same time in a gas burrette. The data for the three types of electrodes is shown in Table III. It was found that the silver mesh does not discharge the zinc electrodes. However, the silver powder and the silver spinel electrodes when connected to the zinc electrode discharge it at a rapid rate.

The non-linear resistance of the diode may be used to block self-discharge current but permit the passage of the recombination current. Such a procedure is described in Patent No. 3080440 by P. Ruetschi of the Electric Storage Battery Company. By proper choice of diode, it should be possible to use an auxiliary electrode so that the self-discharge current will be in the micro-ampere range at a potential of half a volt, while the current would be in the ampere range to near a volt when the voltage climbed due to the recombination potential.

To overcome the discharging problem, the silver spinel electrode was connected to the zinc electrode through 1N457 and 1N198 diodes. The data with these diodes is presented in Table III. With this procedure, little gassing and discharge of zinc electrode would occur. However, when oxygen was introduced into the system, current would pass through this diode, showing recombination of oxygen gas. By use of a diode, it is possible to use an auxiliary electrode which connected directly would discharge the cell.

2.2.2. Oxygen Recombination Studies

These studies are being made with test cells composed of auxiliary electrodes of silver mesh, silver powder and silver spinel and either cadmium or nickel counter electrodes. During overcharging, the cadmium or nickel electrode generates oxygen which is reacted at the auxiliary electrode. This procedure simulates the effect of the oxygen cycle on cell overcharge.

In the test, electrodes of the same cross-section as the electrodes of the Yardney (Model PM-3) 3 ampere-hour silver zinc cells are used (electrode area 14 cm²). The electrodes were assembled in cells with nylon separators, 40% KOH electrolyte added, the cells charged to complete the charge on the nickel hydroxide electrode, and purged with oxygen. The oxygen pressure was adjusted to 700, 100, 10, and 0 millimeters of Hg. The cells were then charged at 12.5 milliamperes continuously for 48-72 hours. A current of 12.5 milliamperes represents a C/20 charge rate. This procedure was repeated for 25 milliamperes and 50 milliamperes.

TABLE III

Self-Discharge of Zinc Electrodes Electrode area - 14 cm²

At 12.5 milliamperes, electrodes of silver mesh and silver powder did not recombine gas at a rate rapid enough to keep the pressure in the system from rising. With a silver spinel electrode, no pressure buildup occurred except at the 50 milliampere rate but not at a very rapid rate. At the end of the test, the pressure in the system would drop, presumably due to adsorption of oxygen by the spinel electrodes. These results indicate that these spinel electrodes may be used for oxygen recombination to a charging rate of at least C/10.

2.2.3. Hydrogen Recombination Studies

Conventional fuel cell hydrogen electrodes and silver modified fuel cell hydrogen electrodes were used in a manner analogous to the one described in the previous section to prove their effectiveness in hydrogen recombination. However, due to the inability to obtain proper catalyst activity, the results obtained with these electrodes were not conclusive.

These electrode materials had previously worked very satisfactorily on hydrogen recombination on both fuel cells and batteries. For this reason, it is believed that the failure of the cells is due to catalyst inactivity rather than improper electrode mechanism. These tests will be re-run in the next quarter with new electrodes.

2.3 Hydrogen Combination Cell

The objective of this task is to assess the feasibility of the electrochemically combining hydrogen evolved from silverzinc satellite primary power supply system. The nominal combination capacity is to be 200 cc $\rm H_2/hour$. This function is presently performed by absorption of hydrogen on palladium or venting. The primary goal is to reduce the weight of the system. The absorption capacity of palladium at ambient temperature and 1 atmosphere partial pressure of hydrogen is approximately $\rm lg~H_2/177g~Pd$.

Copper II oxide appears to be an attractive cathode material from the standpoint of the electrochemical reactivity and mass equivalent weight. The equivalent weight of copper II oxide is 40 g/lg hydrogen.

Test cells using commercially available copper oxide electrodes (McGraw-Edison Alkaline Battery Division) and two types of hydrogen electrodes were made and tested over a range of current densities. The hydrogen electrodes were: (1) auxiliary type

electrodes using platinum catalysts and (2) various thicknesses of rolled palladium-silver alloy sheets. The cells used in this work had a hydrogen electrode area of 6.25 in² and copper oxide electrodes of the same area weighing 90 grams.

The palladium-silver alloy 1 mil thick hydrogen electrode produced a current of 1 milliampere at 0.1 volts. Thicker electrodes (4 mils thick) produced no measurable current. When thinner material was used (0.2 mil, 0.1 mil) the current produced by the cell was much lower. This data is shown on Figure I. It is, therefore, believed that the high current obtained with the first cell was not characteristic of the electrode material but due to some irregularities in the electrode material, such as fissures or pin holes. From the results obtained, it was decided that the palladium-silver alloy is not a practical electrode material.

Some conventional electrodes containing nickel, platinum and palladium were tried as hydrogen electrodes. The current obtained with these electrodes was in the range of 10 milliamperes at 0.4 volts. The results with these electrodes are shown on Figure II. From the results, it may be seen that the conventional electrode material is practical for this use.

One of the difficulties with this cell construction is that the auxiliary type of hydrogen electrode leaks electrolyte. This loss of electrolyte could result in subsequent failure of the cell. To prevent this electrolyte loss, polyethylene film 1 mil thick was placed approximately 1/4" from the hydrogen electrode. In this manner, the electrolyte is restrained in the cell volume. As shown in Figure II, the membrane did not affect the cell operation, since the polyethylene film is very permeable to hydrogen.

An additional benefit of this cell seems to be the fact that it is also capable of recombining oxygen; this cell can operate in an air atmosphere. The action seems to be the reverse of the one for hydrogen recombination:

$$O_2 + 2 H_2O + 4 e \longrightarrow 40H^ 2Cu + 40H^- \longrightarrow 2CuO + 2H_2O + 4 e$$

One of the above cells was sent to NASA-Goddard for further evaluation. Another of these cells is being tested continually to determine its long-term operation characteristics.

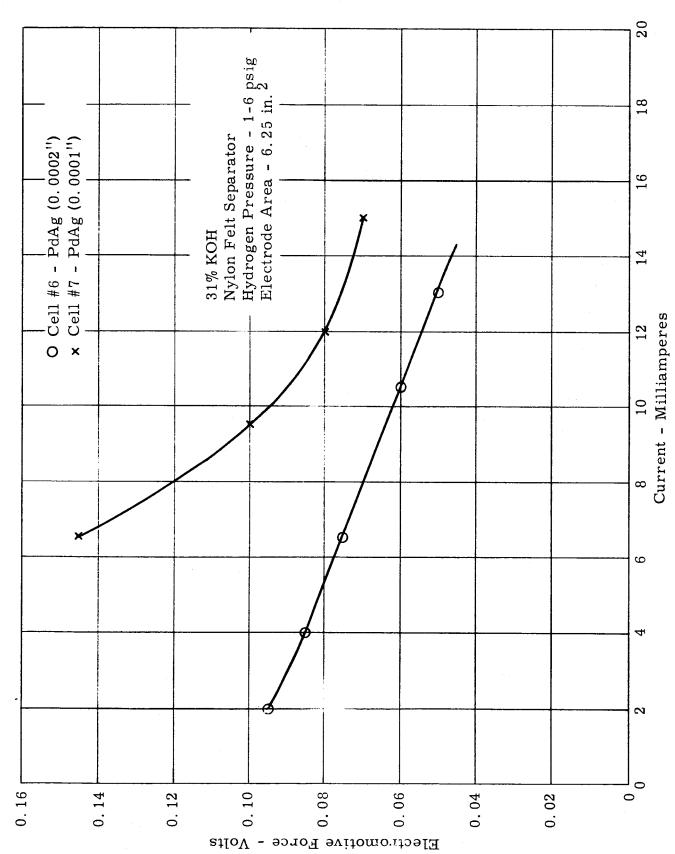


Figure I. Polarization Curves - Hydrogen Combination Cell CuO Versus Pd-Ag Foil.

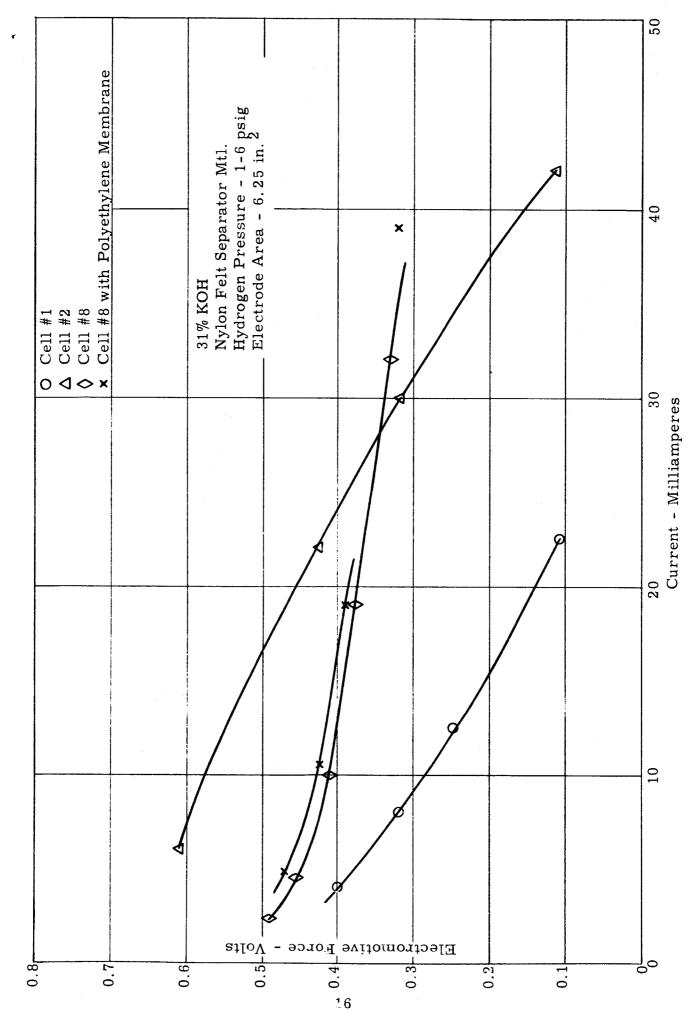


Figure II. Polarization Curves - Hydrogen Combination Cell CuO Versus Pt.-Pd Gas Diffusion Electrode.

3.0 PROGRAM FOR NEXT QUARTER

Testing of the silver-cadmium cells in the continuous overcharge and cyclic modes will be continued.

Additional design data for auxiliary electrodes for recombining hydrogen and oxygen is silver-zinc cells will be obtained. Silver-zinc test cells containing these auxiliary electrodes will be constructed and tested.

Testing of the copper oxide-hydrogen combination cell will be continued.